Specification Shaw Proposal Corrections

U.S. PATENT Docket No. 45074.32

# DETERMINATION OF OIL AND WATER COMPOSITIONS OF OIL/WATER EMULSIONS USING LOW FIELD NMR RELAXOMETRY

Inventors: Mirotchnik, Konstantin; Allsopp, Kevin; Kantzas, Apostolos, Marentette Daniel F.

10 Assignee: University Technologies International Inc.

## PRIORITY CLAIM

5

15 1

25

30

This application claims the priority benefit of Canadian Patent Application No. 2,342,007 filed on March 26, 2001 as file no. 45074.9 and entitled Determination of Oil and Water Compositions of Oil/Water Emulsions Using Low Field NMR Relaxometry.

#### FIELD OF THE INVENTION

The present invention relates to methods and apparatuses for determining oil and water compositions of heavy oil/water emulsions using low field NMR relaxometry.

## **BACKGROUND OF THE INVENTION**

Low field Nuclear Magnetic Resonance (NMR) relaxometry techniques have been developed in the laboratory to enhance and support comparable NMR logging tools that are currently used downhole. Low field NMR relaxometry involves relaxometers operating at about 2 MHz or less. Low field NMR relaxometry has shown that discrimination of water and oil saturation in core and ore can be easily determined. In such cases the NMR can detect the total water weight fraction and the total oil weight fraction, the viscosity of the oil, the amount of bound or mobile water and the amount of mobile or bound oil.

One particular problem is the determination of oil and water content of specific hydrocarbon streams. Of particular interest are the streams that contain heavy oil in

emulsified fluids (water-in-oil or oil-in-water emulsions) which are currently very common in thermal production operations and are very difficult to handle. Test separators are currently used as the standard way of measuring the flow of thermally produced wells such as cyclic steam stimulation (CSS), steam assisted gravity drainage (SAGD) and steam flooding wells. The test separators are inherently incapable of measuring emulsified flow. Other probe-type devices suffer from inaccuracies related to the presence of solids or gas, salinity, temperature, velocity, emulsion type, and range of cut.

Therefore, there is a need in the art for methods and apparatuses to discriminate quickly, accurately and precisely the amount of heavy oil or bitumen and water in an emulsified fluid stream.

## SUMMARY OF THE INVENTION

15

10

The present invention is based on the discovery that the NMR spectra of an emulsified mixture of heavy oil or bitumen and water consists of two sets of T<sub>2</sub> relaxation peaks. At the specific temperature of 30°C, the water peaks are typically in the range of 10 to 3000 milliseconds while the oil/bitumen peaks are typically in the range of 0.2 to 10.0 milliseconds. The ranges of these peaks may be affected by the degree of emulsification or separation of the hydrocarbon and aqueous phases, the temperature and the presence of additives. The spectrum of the oil/bitumen component diminishes at lower temperatures and may not be completely recovered at relatively lower temperatures.

25

30

Therefore, in one aspect of the invention, there is provided a method of determining the oil fraction of a fluid emulsion comprising heavy oil/bitumen and water by direct measurement comprising the steps of:

## (a) providing a low field NMR relaxometer:

- (b) measuring and recording the T<sub>2</sub> relaxation spectrum of the emulsion at a temperature allowing recovery of the T<sub>2</sub> spectrum of the heavy oil/bitumen, substantially separate from a T<sub>2</sub> water peak;
- (c) determining a distinguishing T<sub>2</sub> cutoff value;
- (d) measuring the total amplitude  $(A_{oil})$  of the spectrum at  $T_2$  times less than and equal to the  $T_2$  cutoff value:
- (e) converting A<sub>oil</sub> to a weight value by dividing A<sub>oil</sub> by the

  amplitude index of an oil standard (AI<sub>oil</sub>).of known weight; and
- (f) using the weight value to determine the oil fraction of the fluid emulsion.

In another aspect, the invention comprises a method of determining the water fraction of a fluid emulsion comprising heavy oil/bitumen and water by direct measurement comprising the steps of:

(a) providing a low field NMR relaxometer:

10

15

20

25

- (b) measuring and recording the T<sub>2</sub> relaxation spectrum of the emulsion;
- (c) determining a distinguishing T2 cutoff value;
- (d) measuring the total amplitude (A<sub>w</sub>) of the spectrum at T<sub>2</sub> times greater than the T<sub>2</sub> cutoff value:
- (e) converting A<sub>w</sub> to a weight value by dividing A<sub>w</sub> by the

  amplitude index of a water standard (AI<sub>w</sub>).of known weight;

  and
- (f) (f) using the weight value to determine the water fraction.

In another aspect, the invention comprises an apparatus for determining <u>by</u> <u>direct measurement</u> the oil fraction of a flowing fluid emulsion comprising heavy oil <u>bitumen</u> and water comprising:

- (a) a low field NMR relaxometer having a NMR magnet positioned in proximity to a channel through which the emulsion flows, said relaxometer for measuring the T<sub>2</sub> spectrum of a the sample at a temperature allowing recovery of the T<sub>2</sub> spectrum of the heavy oil/bitumen, substantially separate from a T<sub>2</sub> water peak;
- (b) means for identifying a distinguishing T2 cutoff value:

10

15

20

- (c) means connected to the relaxometer for measuring total T<sub>2</sub> amplitude below a the T<sub>2</sub> cutoff value, wherein a substantial portion of the spectrum attributable to the oil is at T<sub>2</sub> values less than or equal to the T<sub>2</sub> cutoff value;
- (d) means for converting the total T<sub>2</sub> amplitude value to a weight value; and
- (e) means for determining the weight value to determine the oil fraction of the fluid emulsion.

In yet another aspect, the invention comprises an apparatus for determining by direct measurement the oil fraction of a fluid emulsion comprising heavy oil/bitumen and water comprising:

- (d) means for obtaining a sample of the emulsion:
- 25 (p) a low field NMR relaxometer for measuring the T<sub>2</sub> spectrum of the sample at a temperature allowing recovery of the T<sub>2</sub>

spectrum of the heavy oil/bitumen, substantially separate from a T<sub>2</sub> water peak;

- (c) means for identifying a distinguishing T2 cutoff value;
- (d) means connected to the NMR relaxometer for measuring total

  T<sub>2</sub> amplitude below a the T<sub>2</sub> cutoff value, wherein a substantial

  portion of the spectrum attributable to the oil is at T<sub>2</sub> values less
  than or equal to the T<sub>2</sub> cutoff value;
- (e) means for converting the total T<sub>2</sub> amplitude value to a weight value; and
- (f) means for determining the weight value to determine the oil fraction of the fluid emulsion.

In another aspect, the invention comprises a <u>method of determining by direct</u>

<u>measurement the oil fraction and water fraction of a fluid emulsion</u>

<u>comprising heavy oil/bitumen and water comprising the steps of:</u>

(a) providing a low field NMR relaxometer;

10

20

- (b) measuring and recording the T<sub>2</sub> relaxation spectrum of the emulsion at a temperature allowing recovery of the T<sub>2</sub> spectrum of the heavy oil/bitumen substantially separate from a T<sub>2</sub> water peak;
- (c) determining a distinguishing T<sub>2</sub> cutoff value;
- (d) measuring the total amplitude  $(A_{oil})$  f the spectrum at  $T_2$  times less than and equal to the  $T_2$  cutoff value;
- 25 (e) converting A<sub>oil</sub> to a weight value by dividing A<sub>oil</sub> by the
  amplitude index of an oil standard (AI<sub>oil</sub>) of known weight;

- (f) measuring the total amplitude  $(A_w)$  of the spectrum at  $T_2$  times greater than the  $T_2$  cutoff value;
- (g) converting A<sub>w</sub> to a weight value by dividing A<sub>w</sub> by the

  amplitude index of a water standard (AI<sub>w</sub>).of known weight;

  and
- (h) using the oil weight value and the water weight value to determine the oil fraction and water fraction respectively.

## 10 BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described by way of exemplary embodiments with reference to the accompanying drawings. In the drawings:

Figure 1 shows a typical NMR T<sub>2</sub> spectra from two different emulsions.

Figure 2 shows the comparison of NMR predicted water content vs. Dean-Stark measured water content for three different batches of samples form reservoir 1.

Figure 3 shows the same results as Figure 2 but are grouped and the trend-line is plotted.

Figure 4 shows the comparison of the NMR predicted data and the Dean-Stark measurement data for three samples of reservoir 2.

Figure 5 shows a comparison of the results of reservoir 1 and reservoir 2.

Figure 6 shows the same results as Figure but are grouped and the common trendline is plotted.

25

15

## BENNETT JONES

## FAX MESSAGE

Examiner Tiffany Fetzner U.S. Patent and Trademark Office Bennett Jones LLP 1000 ATCO Centre 10035 - 105 Street Edmonton Alberta T5J 3T2

> Tel 780.917.5231 Fax 780.421.7951

571-273-2241 FAX No. PHONE NO. April 30, 2004 DATE

Edward (Ted) You FROM 45074.32 LAWYER NO FILE NO 807 Original Status - Retained on File

This is the first page of \$ 7

If all pages not received, call 780.421.8133 for assistance.

This facsimile was successfully transmitted at:

#### MESSAGE

Application No. 09/852,339 May 11, 2001 Filing Date, Mirtochnik Inventor:

Group Art Unit: 2859

FETZNER, Tiffany A. Examiner:

Docket No.

45074.32

Further to our telephone discussions this morning, please find enclosed proposed amendments to the specification. All additions to the spec are in 14 pt font and underlined.

Edward Yoo, 41435

THIS MESSAGE IS INTENDED ONLY FOR THE USE OF THE ADDRESSEE AND MAY CONTAIN INFORMATION THAT IS PRIVILEGED AND CONFIDENTIAL. IF YOU ARE NOT THE INTENDED RECIPIENT, OR THE EMPLOYEE RESPONSIBLE FOR DELIVERING THE MESSAGE TO THE INTENDED RECIPIENT, YOU ARE HEREBY NOTIFIED THAT ANY DISSEMINATION OF THIS COMMUNICATION IS STRICTLY PROMIBITED. IF YOU HAVE RECEIVED THIS COMMUNICATION IN ERROR, PLEASE NOTIFY US IMMEDIATELY BY TELEPHONE. THANK YOU.

CALGARY . EDMONTON . TORONTO